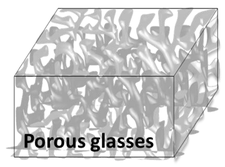
Nanoscale Characterization of Amine-Epoxy Interphase in 3D Confinement network (porous glass)

Marlena Filimon[1], Jörg Schmauch[2], Roland Sanctuary[1]

[1] Laboratory for the Physics of Advanced Materials, University of Luxembourg, 162a avenue de la Faïencerie, Luxembourg
[2] Department of Technical Physics, University of Saarland, Saarbrücken, Germany
marlena.filimon@uni.lu

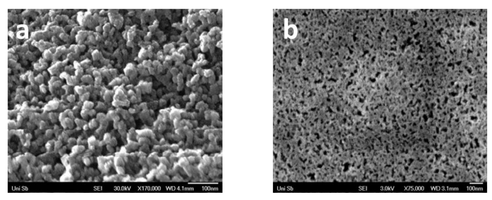
Many aspects of polymer research and application are directly connected with surface and interface phenomena occurring when polymers are brought into contact with substrates made of another kind of material (e.g. metals, nanoparticles, etc.).
Generally, interphases emerge between polymer matrix and substrate. These are regions with morphologies and properties differing from those of polymer and substrate. While curing a thermoset, at least two different types of monomers react to form a high molecular weight polymer network. The composition of the mixture of reactance can be altered near substrate in contact with thermoset components. In this poster, we present some preliminary results obtained for interphases which appear while curing an amine-epoxy thermoset in contact with a porous glass (pore size ~20 nm).
As tools for investigating the interphases, we exploited Scanning Electron Microscopy (SEM) in addition with Energy-Dispersive X-ray Spectroscopy (EDS) and Tapping-Mode Atomic Force Microscopy (TM - AFM).

Materials &Methods



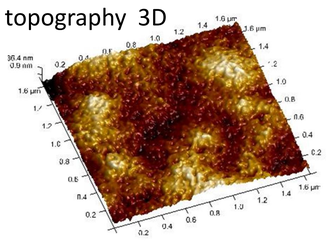
Porous glass, pore size ~ 20 nm;
- sol-gel preparation, porosity (~45%)
- "flat surface" : 1) mechanically
2) ion beam polisher

1) Characterization of empty porous glass (SEM & AFM)

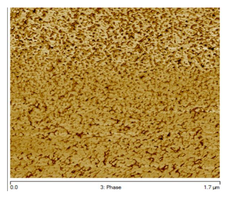


SEM micrographs: a) unpolished; b) polished

topography 3D



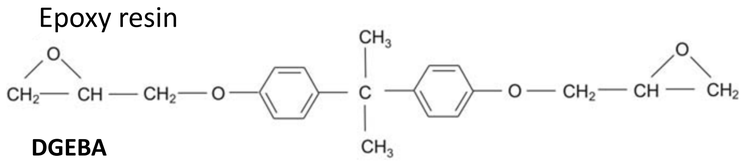
phase



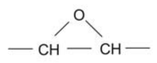
AFM images (for polished porous glass)

2) Curing epoxy (amine-epoxy system)

Epoxy resin



DGEBA
(diglycidyl ether of bisphenol A) D.E.R. 331 Dow Plastics

reactive epoxy (oxirane group) 

Hardener

$H_2N - CH_2 - CH_2 - NH - CH_2 - NH_2$

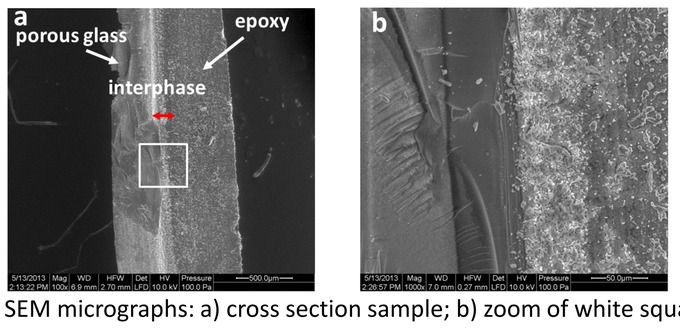
DETA (diethylenetriamine)

3) Interphase: amine-epoxy on porous glass

- low viscosity liquid mixture of epoxy resin & the amine-curing agent - in contact with a solid surface

- when an epoxy resin is cured with an amine hardener in contact with porous glass

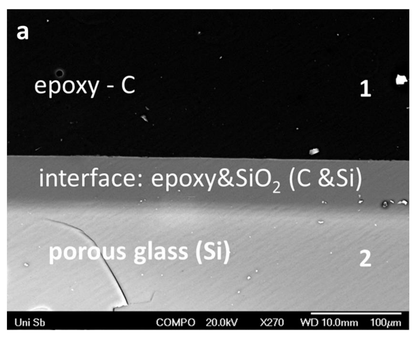
→ one component can be preferentially adsorbed onto the surface.



SEM micrographs: a) cross section sample; b) zoom of white square

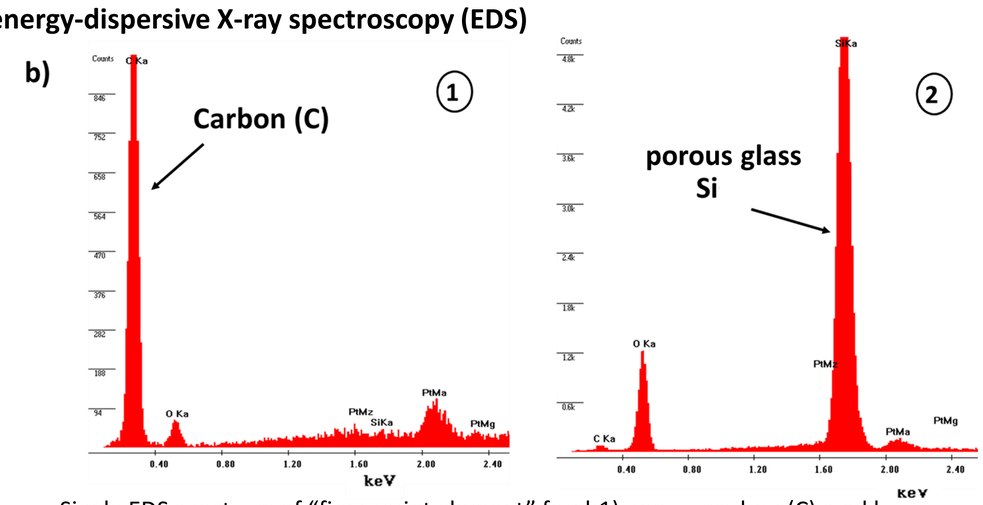
Results & Discussions

1) Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS)



backscattered electron analysis – composition (due to atomic number) ; dark region- correspond to carbon (C) and bright region to silica (Si)

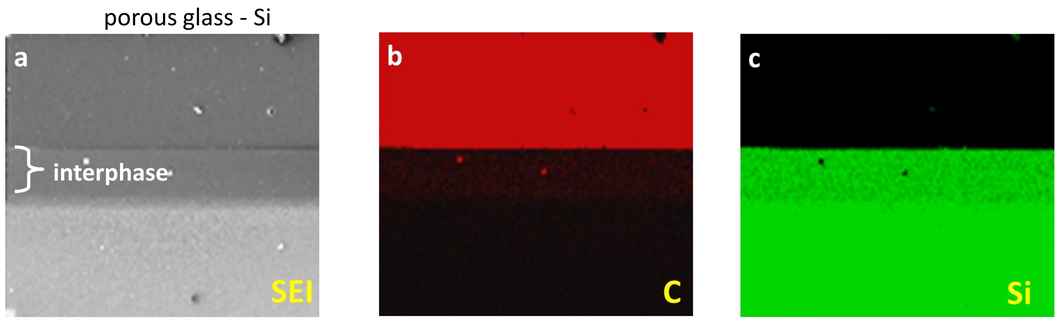
b)



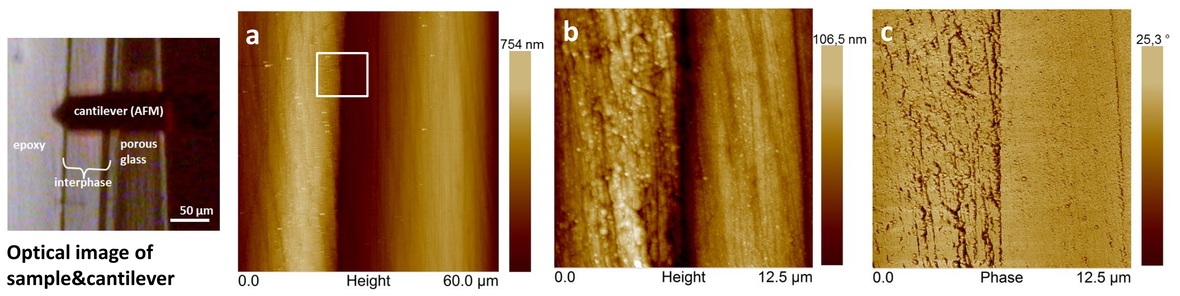
Single EDS spectrum of "fingerprint element" for: b1) epoxy - carbon (C) and b2) porous glass - Si

EDS "chemical" mapping →
(C and Si- reference) 128 /128 spectra
a) secondary electron image-as reference
b) "Chemical" map – follow C
c) "Chemical" map – follow Si

a clear interphase ~50 µm is observed

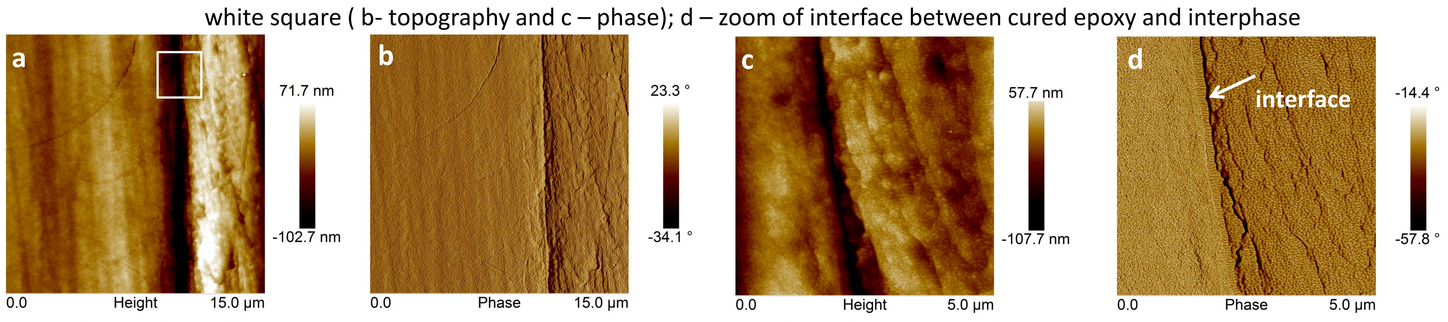


2) Atomic force microscopy (AFM) measurements

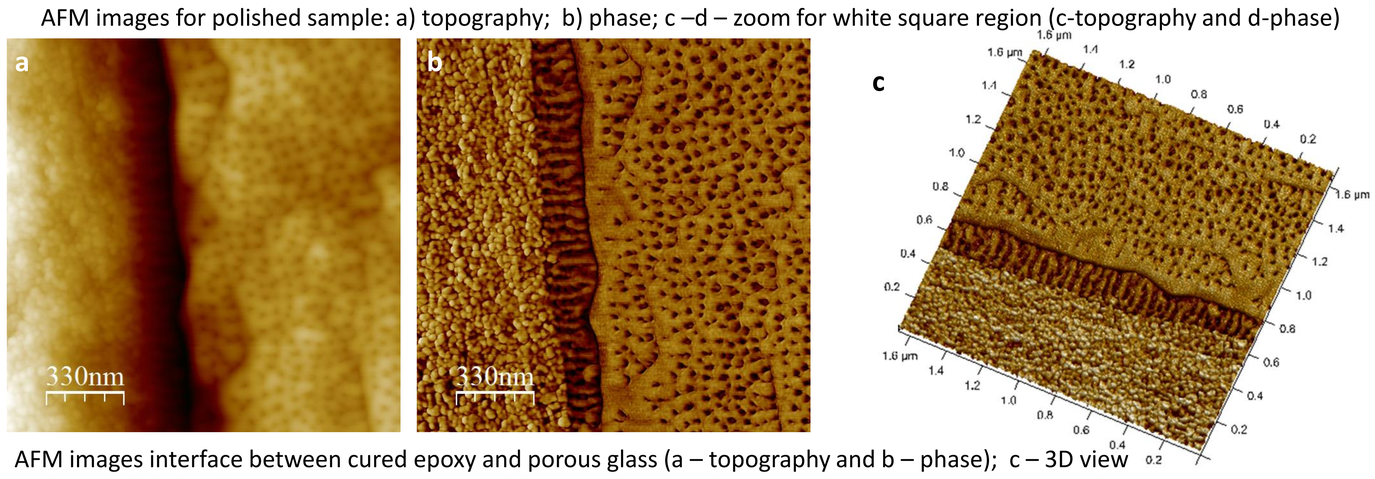


Optical image of sample&cantilever

AFM images of cross section sample unpolished: cured amine-epoxy&porous glass: a) topography overview; b, c – zoom of white square (b- topography and c – phase); d – zoom of interface between cured epoxy and interphase



AFM images for polished sample: a) topography; b) phase; c –d – zoom for white square region (c-topography and d-phase)



AFM images interface between cured epoxy and porous glass (a – topography and b – phase); c – 3D view

A clear well defined new structure is observed (~100 nm) at interface: cured amine-epoxy & porous glass